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Final Report on Effect of Irradiation on Microstructure and Mechanical Properties of Copper and Copper Alloys

(ITER R & D Task No. T13 and T213)

B.N. Singh

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April 1998

Abstract The present note is the final report on investigations carried out under ITER Task No. T13 (1994). Most of the results of these investigations have been published in the open literature either as articles or reports. A list of the appropriate references are given. Results that have been presented at various meetings but have not been published are summarized in the present document. In addition, the present report also clarifies the status of the deliverables in the ITER Task No. T213 (1995). Finally, the main conclusions emerging from these investigations are highlighted.

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1 Introduction

One of the direct consequences of defect accumulation at lower temperatures ($\leq$0.3 T_m) is a substantial increase in the yield strength and a drastic decrease in the ductility already at rather low doses (~0.1 dpa). This is commonly known as low temperature embrittlement and is a matter of a serious concern regarding the performance and lifetime of components exposed to strong irradiation environment (e.g. first wall and divertor). At higher irradiation temperatures ($\geq$0.35 T_m), the defect accumulation, particularly in fcc metals and alloys occurs in the form of voids. This gives rise to the problem of volumetric swelling which may limit the application of materials in components exposed to an intense flux of 14 MeV neutrons in a fusion reactor.

At present copper alloys (Cu-Al_2O_3, CuCrZr and CuNiBe) are being considered as potential candidate materials for the first wall and divertor components of ITER (International Thermonuclear Experimental Reactor). During operation, these components may experience temperatures in the range of about 100 to 350°C. The amount of available experimental results on the evolution of defect microstructure and its impact on the deformation behaviour of these copper alloys, on the other hand, is rather limited particularly for temperatures at which the copper alloys may be employed in service. The present investigations are a part of the ITER research and development programme and cover the expected temperature range of ~50 to 350°C. In the following, the results on the microstructural evolution and the mechanical properties of copper and copper alloys (CuCrZr, CuNiBe and Cu-Al_2O_3) irradiated at temperatures in the range of 50 to 350°C are summarized. The main conclusions and implications are highlighted in section 5.

2 Materials and Experimental Procedure

The materials used in the present investigations were thin (0.3 mm) sheets of oxygen free high conductivity (OFHC) copper and copper alloys (CuCrZr, CuNiBe and dispersion strengthened (DS) Cu-Al_2O_3). The OFHC copper, CuCrZr and CuNiBe alloys were supplied by Tréfimétaux (France) and the DS copper (GlidCop CuAl25) was supplied by SCM Metals (USA). The chemical composition of these alloys is listed in Table 1. Henceforth, the DS copper (Cu-Al_2O_3) will be referred to as CuAl25.

Table 1. Chemical Composition

CFHC-Cu:	Cu - 10, 3 <1 and <1 ppm of Ag, Si, Fe and Mg, respectively
CuCrZr:	Cu - 0.8% Cr, 0.07% Zr, 0.01% Si
CuNiBe:	Cu - 1.75% Ni, 0.45% Be
CuAl25	Cu - 0.25% Al as oxide particles (0.46% Al_2O_3)

Details of the specimen geometry, thermomechanical treatments prior to irradiations, irradiation procedure and conditions, and microstructural characterizations are described in various publications (e.g. [1,2,6,7,12].

3 Results (Task No. T13 (1994))

Part 1

(a) Results on hardness and tensile properties of OFHC-Cu, $CuAl_2O_3$ and CuCrZr irradiated at 50°C:
  (i) results are described and discussed in [1-4],
  (ii) corresponding experiments were carried out on European CuNiBe alloy (in the prime aged conditions) and the results are included in [1-4].

(b) Results on hardness and tensile properties of OFHC-Cu, Cu-Al_2O_3 and CuCrZr irradiated at 250, 350 and 450°C:
  (i) some of the results are given in [5-8],
  (ii) the unpublished results are presented in Annex 1-3.

(c) Results on the low cycle fatigue behaviour of OFHC-copper, Cu-Al_2O_3 and CuCrZr irradiated at 50°C:
  (i) prior to investigating the effect of irradiation, the effect of specimen size on the low cycle fatigue behaviour was investigated. This step was necessary since it was unpractical to carry out irradiation tests on the ASTM standard size specimens. Results of these investigations are reported in [3, 9-11],
  (ii) the results of the low cycle fatigue tests on copper and copper alloys irradiated at 50°C and tested at 50°C are described and discussed in [4,8,12].

(d) Results on the low cycle fatigue behaviour of OFHC-Cu, Cu-Al_2O_3 and CuCrZr at 250, 350 and 450°C.
  (i) these high temperature fatigue tests were postponed because of the urgency of Screening Experiments,
  (ii) the low cycle fatigue behaviour of these alloys, however, has been investigated on specimens irradiated at 100°C and the corresponding unirradiated specimens. Results are described and discussed in [4,8,12].
  (iii) low cycle fatigue testing of specimens irradiated at 250 and 350°C is in progress,
  (iv) additional specimens of OFHC-Cu and CuAl-25 were irradiated (during 1994) in EBR-II at ~375°C to doses in the range of 1.1 to 6.2 dpa. These specimens will be also fatigue tested.

(e) Results on the irradiation induced microstructure in OFHC-Cu, Cu-Al_2O_3 and CuCrZr irradiated at 50, 250, 350 and 450°C:
  (i) results are described and discussed in [1, 2, 5, 6, 7],
  (ii) additional information on the effect of irradiation on void swelling in MARZ-Cu, CuCrZr and Cu-5%Ni irradiated (in the MOTA assembly of the FFTF reactor at Richland) at temperatures in the range of ~420-530 up to dose levels of 31 - 34 dpa is given in [13],
  (iii) microstructural information on unirradiated and irradiated (at 250°C) CuCrZr is given in Table 1 of Annex-II.

Part 2

(a) 600 MeV proton irradiation (in the PIREX facility at PSI) of CuCrZr and Cu-Al_2O_3:

(i) only a limited number of specimens were irradiated before the irradiation programme with 600 MeV protons was terminated.

(ii) tensile results on OFHC-Cu and CuAl-25 irradiated at 75 - 390°C to a dose level of ~0.5 dpa are given in Annex-IV,

(iii) the effect of 600 MeV and 750 MeV proton irradiation on microstructural changes and the irradiation stability of Al_2O_3 particles were investigated and the results are given in [14-17].

(iv) Results on effects of helium implantation rate and temperature on cavity formation are given in [18]. Effect of implanted helium on void swelling is described in [19].

4 Status (deliverables in the Task T213 for 1995)

2.1. (Risø-1): See Annex-II, Figs. 1, 2, 4 and Table 1.

2.2. (Risø-2): See Annex-I (Fig. 5), Annex-II (Fig. 3) and Annex-III (Fig. 3).

2.3. (Risø-3): Risø-R-937 (EN), January 1997 and Risø-R-971 (EN) February 1997.

2.4. (Risø-4): Risø-R-937 (EN), January 1997.

2.5. (Risø-5):
} Risø-R-991 (EN), May 1997.
2.6 (Risø-6):

5 Conclusions

Detailed conclusions based on the results of various investigations are described in the respective publications and will not be repeated here. In the following, however, some of the major conclusions are highlighted.

- The most significant effect of irradiation at low temperatures is a drastic decrease in the uniform elongation of both copper and copper alloys at a dose level as low as ~0.2 dpa. The loss of ductility appears to be related to the intrinsic hardness of the grain interior and not to the grain boundary embrittlement.
- At low temperatures, CuNiBe exhibits better uniform elongation than CuCrZr and CuAl-25.

- The Al_2O_3 particles in the CuAl-25 alloy are found to be reasonably stable against irradiation as well as thermal treatments.
- The precipitates in CuCrZr and particularly in CuNiBe seem to suffer from ballistic resolution. The ballistic resolution and diffusional segregation seem to cause grain boundary embrittlement in CuNiBe at higher irradiation temperature (see results at 250 and 350°C).
- Even though the irradiated copper and copper alloys suffer from a drastic decrease in the uniform elongation, all samples irradiated and tested at lower temperatures fracture in a ductile manner.
- The CuAl-25 exhibits the greatest resistance to radiation-induced changes in the microstructure and the mechanical properties. However, the limited hardening ability of CuAl-25 may have deleterious effects on its fracture toughness behaviour.
- The microstructure of both precipitation hardened (i.e. CuCrZr and CuNiBe) alloys is sensitive to irradiation. This may present problems regarding their performance at higher temperatures and displacement doses.
- During low cycle fatigue tests, both OFHC-Cu and CuCrZr deform inhomogeneously and exhibit extensive necking. The amount of necking observed in the irradiated CuAl-25 specimens, however, is rather small.
- Irradiated specimens, particularly of CuAl-25, exhibit a noticeable improvement in the fatigue performance due to irradiation. This improvement in the case of CuAl-25 alloy is quite significant in specimens irradiated and tested at 100°C.
- The analysis of the present results suggests, however, that the irradiation-induced improvement in the fatigue lifetime observed in the post-irradiation test may not occur during the service condition of ITER where the accumulated damage in one burn-up cycle may not be high enough to decorate the grown-in dislocations (by small SIA loops) and render them immobile. On the other hand, since the mobile dislocations generated during cyclic deformation are quite effective in removing the irradiation-induced defect clusters, the irradiation may not cause deleterious effects on the fatigue lifetime of these materials.
- It is suggested that the post-irradiation fatigue testing of specimens irradiated to low doses (i.e. $\leq 10^{-2}$ dpa) may be useful in verifying the above conclusion, particularly in the case of CuAl-25 alloy.

Acknowledgements

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ANNEX I

Tensile Properties of OFHC-Cu Irradiated at 250, 350 and 450°C with Fission Neutrons

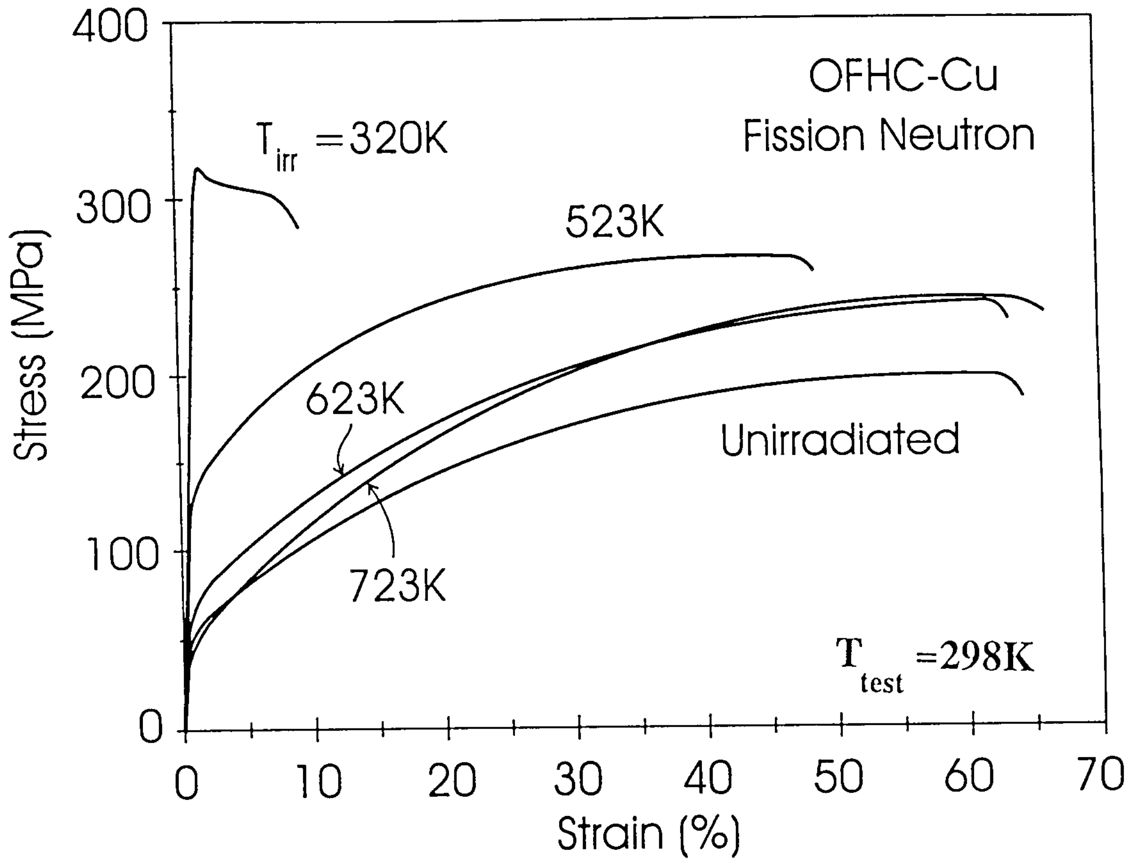

Figure 1. Stress-strain curves for OFHC-copper irradited with fission neutrons at different temperatures to a displacement dose level at ~0.3 dpa. All specimens were tensile tested at room temperature. Note the appearance of plastic instability at 0.3 dpa.

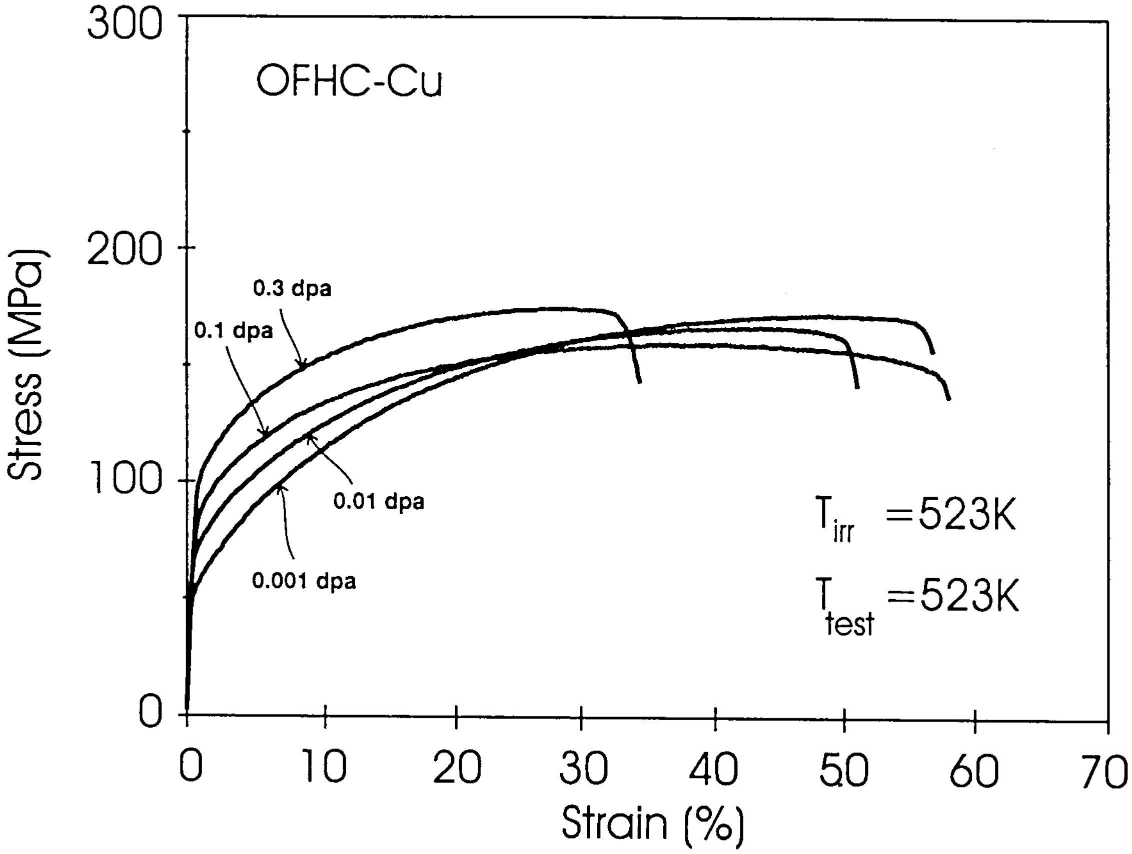

Figure 2. Stress-strain curves for OFHC-copper irradiated with fission neutrons to different displacement dose levels at 523 K and tensile tested at 523 K. Note the absence of plastic instability at 0.3 dpa.

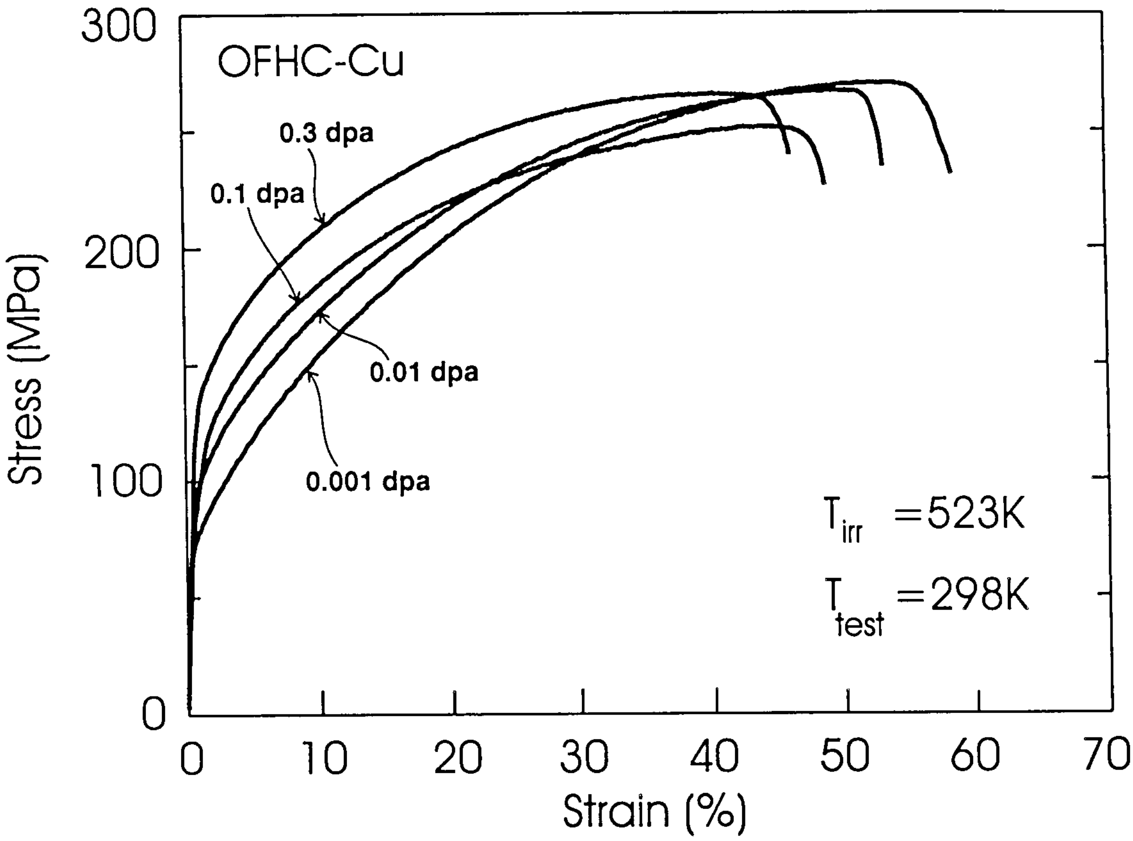

Figure 3. Same as Figure 2 but tensile tested at room temperature. Note the absence of plastic instability at 0.3 dpa.

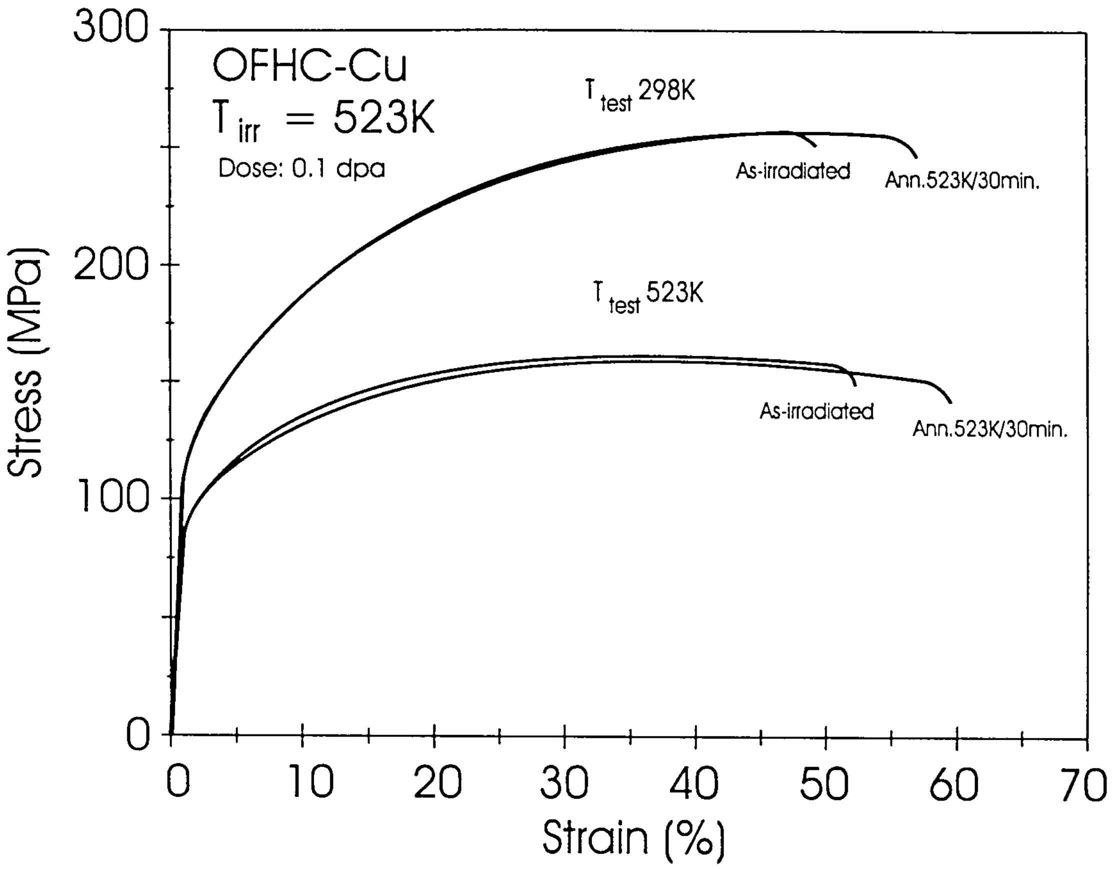

Figure 4. Stress-strain curves for OFHC-copper irradiated at 523 K to ~0.1 dpa and tested at room temperature and 523 K in the as-irradiated condition and after post-irradiation annealing at 523 K for 30 min. The results show that heating up to the tensile test temperature does not affect the final deformation behaviour in any significant way.

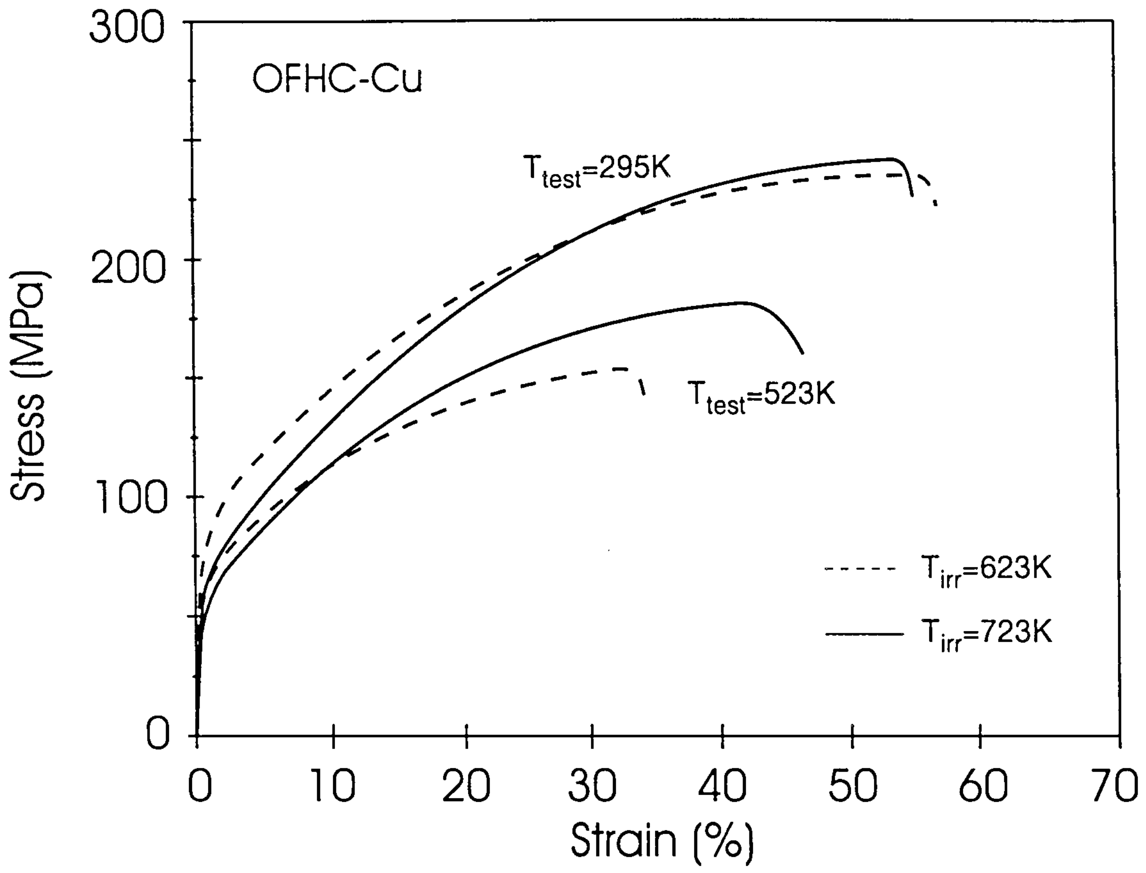

Figure 5. Stress-strain curves for OFHC-copper irradiated to a displacement dose level of 0.3 dpa at 623 and 723 K and tested at 295 K and 523 K.

ANNEX II

Tensile Properties of the Prime Aged CuCrZr Irradiated at 250, 350 and 450°C with Fission Neutrons

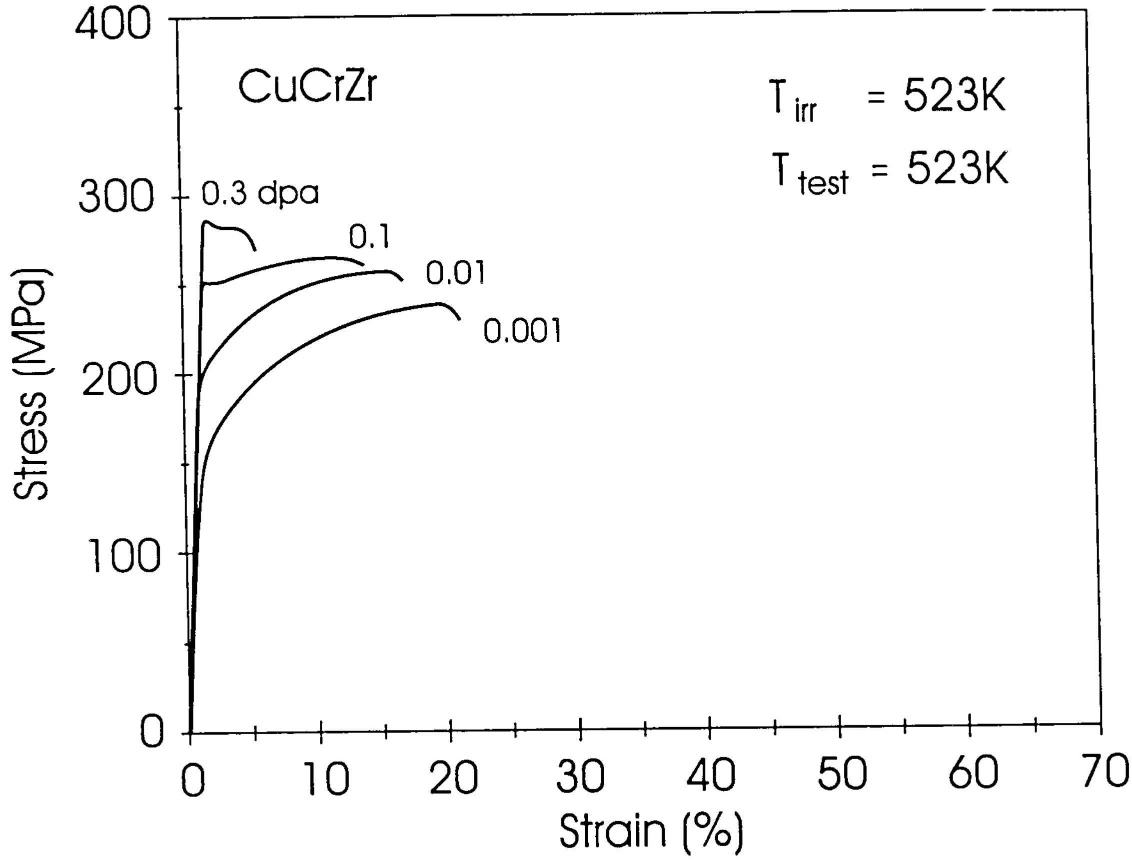

Figure 1. Stress-strain curves for the prime-aged CuCrZr irradiated to different displacement dose levels at 523 K and tensile tested at 523 K.

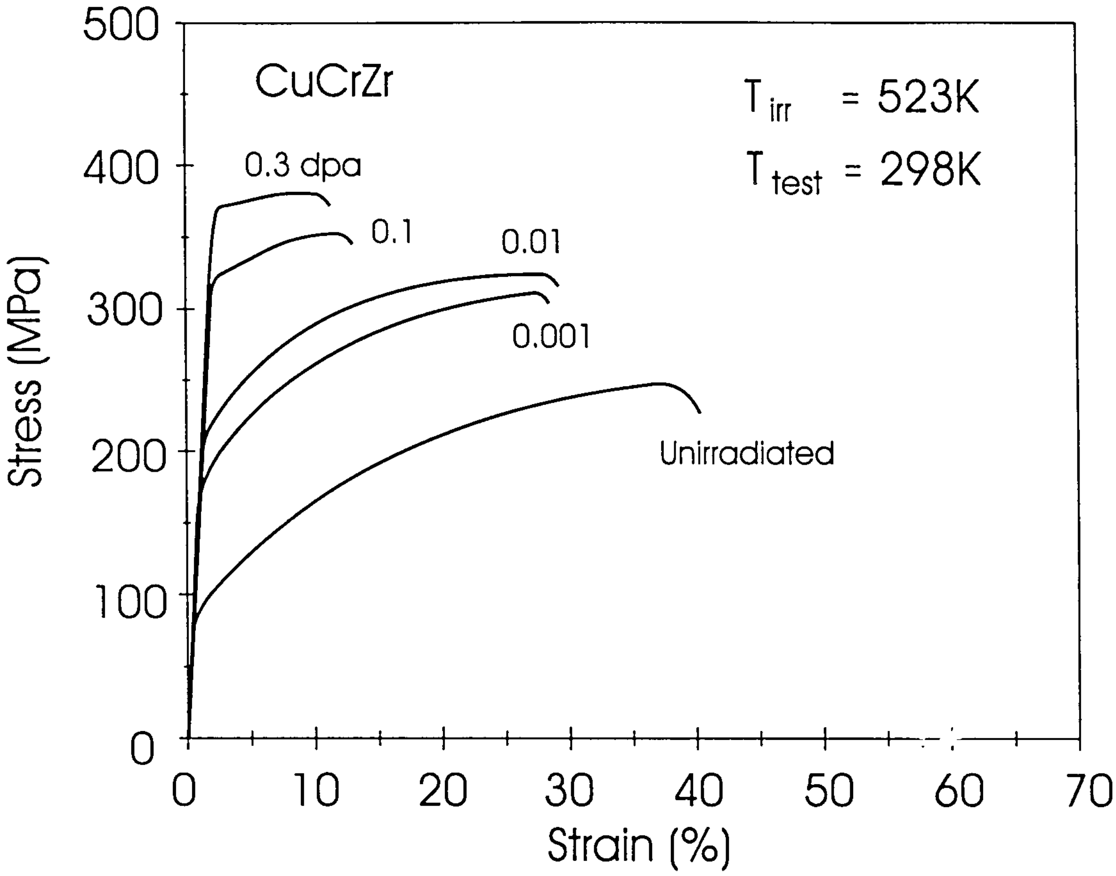

Figure 2. Same as in Figure 1 but tested at room temperature. For comparison, the stress-strain curve for the unirradiated specimen is also shown.

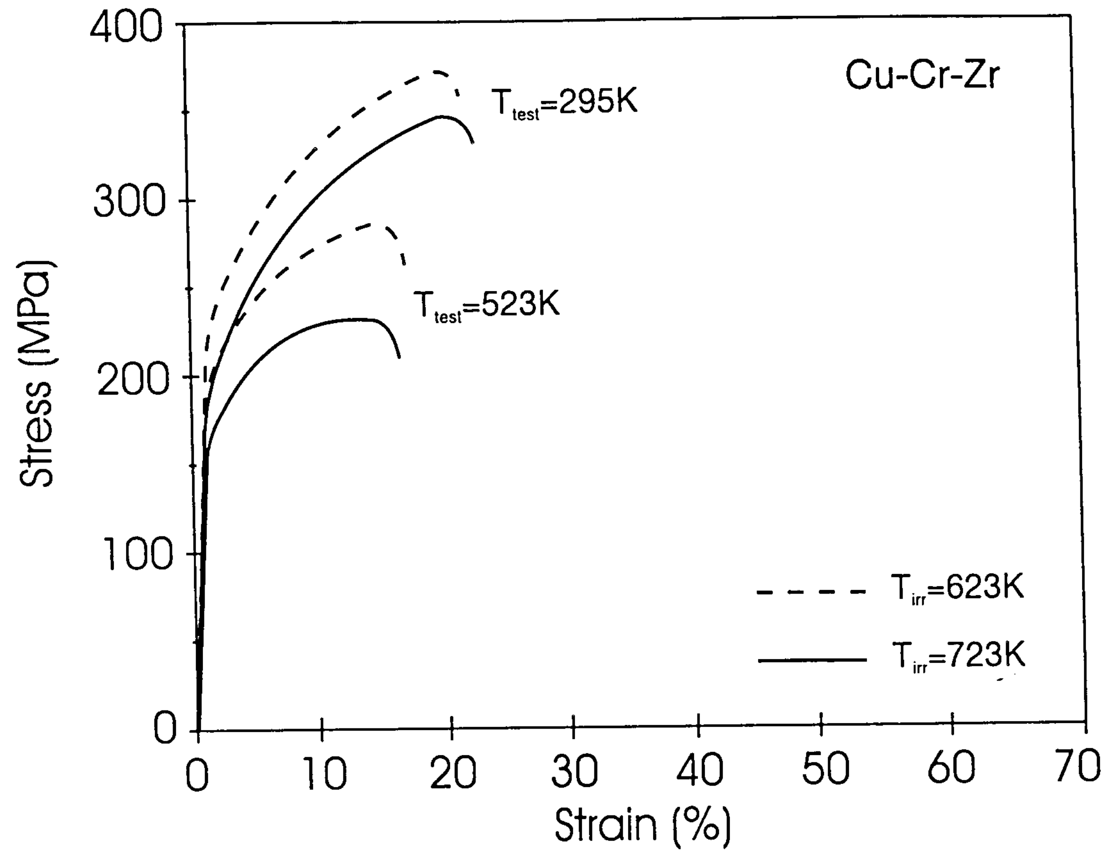

Figure 3. Stress-strain curves for the prime-aged CuCrZr irradiated at 623 and 723 K and tensile tested at room temperature and 523 K.

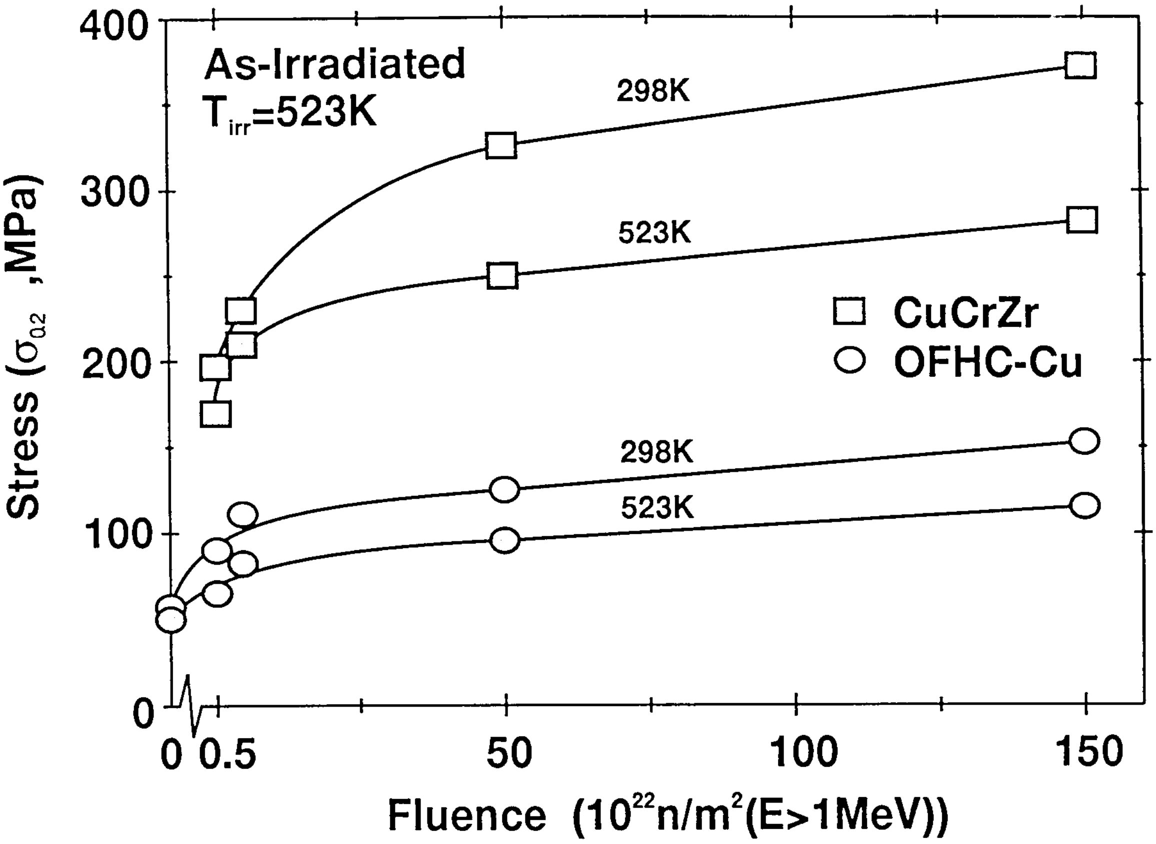

Figure 4. The plastic flow stress ($\sigma_{0.2}$) as a function of neutron fluence for CuCrZr and OFHC-copper irradiated at 523 K and tested at room temperature and 523 K.

Table 1. (Annex II): Defect clusters and precipitates in CuCrZr (prime-aged) in unirradiated and irradiated (at 250°C) conditions

	Unirradiated	0.001 dpa	0.01 dpa	0.1 dpa	0.3 dpa
Cluster Density (m^{-3})	-	2.3×10^{22}	5.7×10^{22}	4.7×10^{23}	3.3×10^{23}
Precipitate Density (m^{-3})	1.5×10^{23}	7.4×10^{22}	8.4×10^{22}	9.6×10^{22}	7.9×10^{22}

ANNEX III

Tensile Properties of Cu-Al_2O_3 (CuAl-25) Irradiated at 250, 350 and 450^{o}C with Fission Neutrons

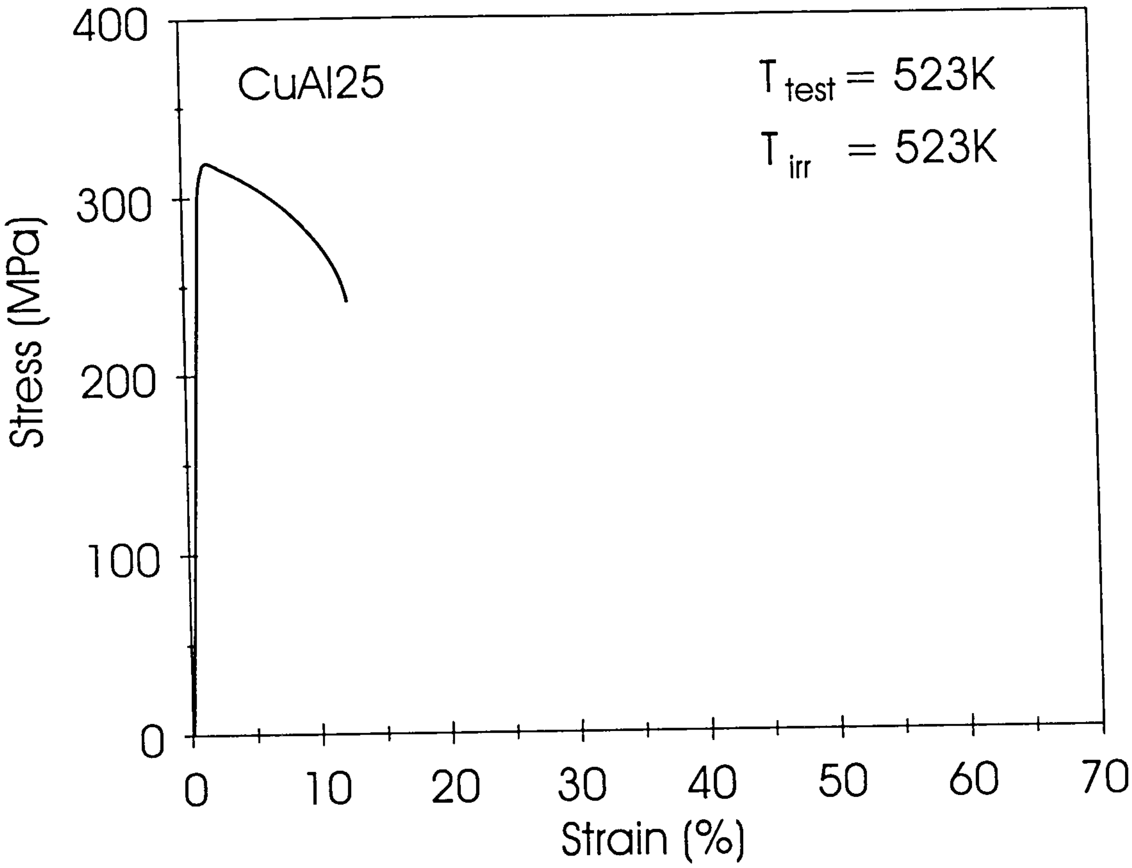

Figure 1. Stress-strain curve for Cu-Al_2O_3 (CuAl-25) irradiated (to 0.3 dpa) and tensile tested at 523 K.

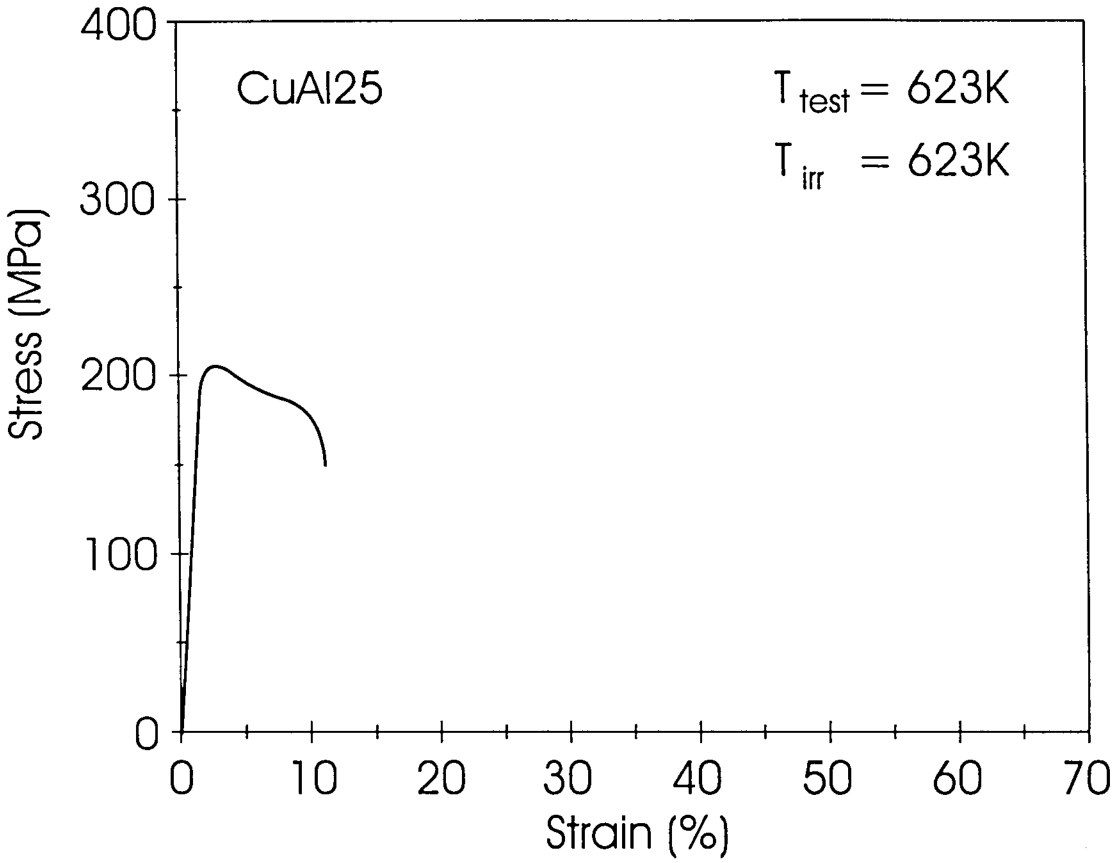

Figure 2. Same as in Figure 1 but irradiated and tensile tested at 623 K.

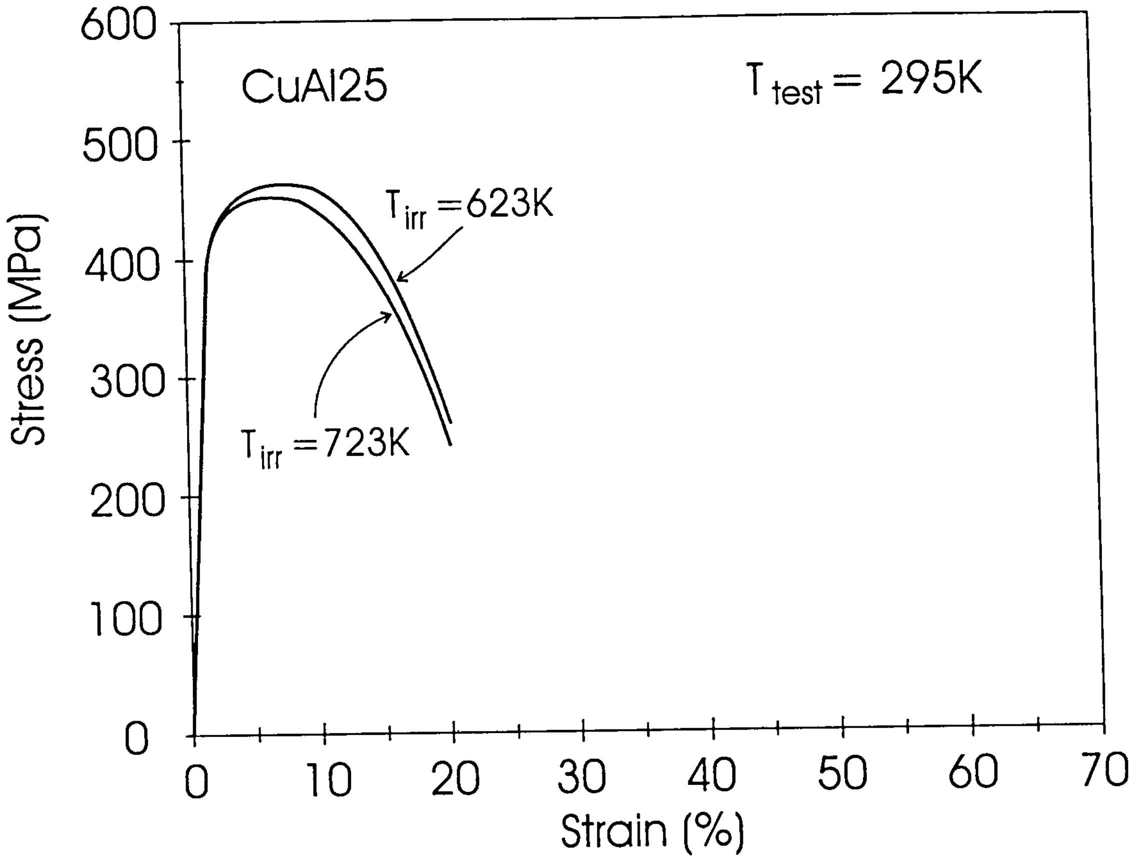

Figure 3. Stress-strain curves for Cu-Al_2O_3 (CuAl-25) irradiated to 0.3 dpa at 623 and 723 K and both tested at room temperature.

ANNEX IV

Tensile Properties of OFHC-Cu and Cu-Al_2O_3 (CuAl-25) Irradiated with 600 MeV protons at 75 - 390°C

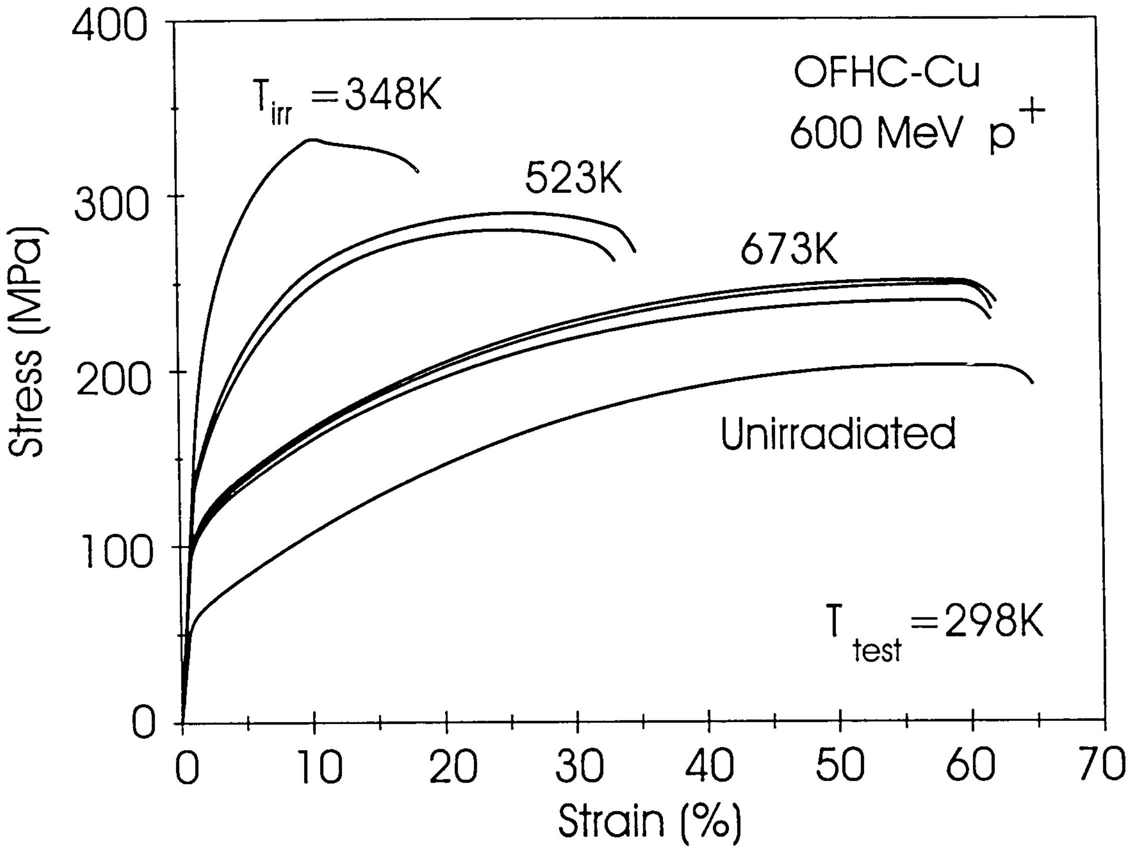

Figure 1. Stress-strain curves for OFHC-copper irradiated with 600 MeV protons at different temperatures to a displacement dose level of ~0.5 dpa and tested at room temperature.

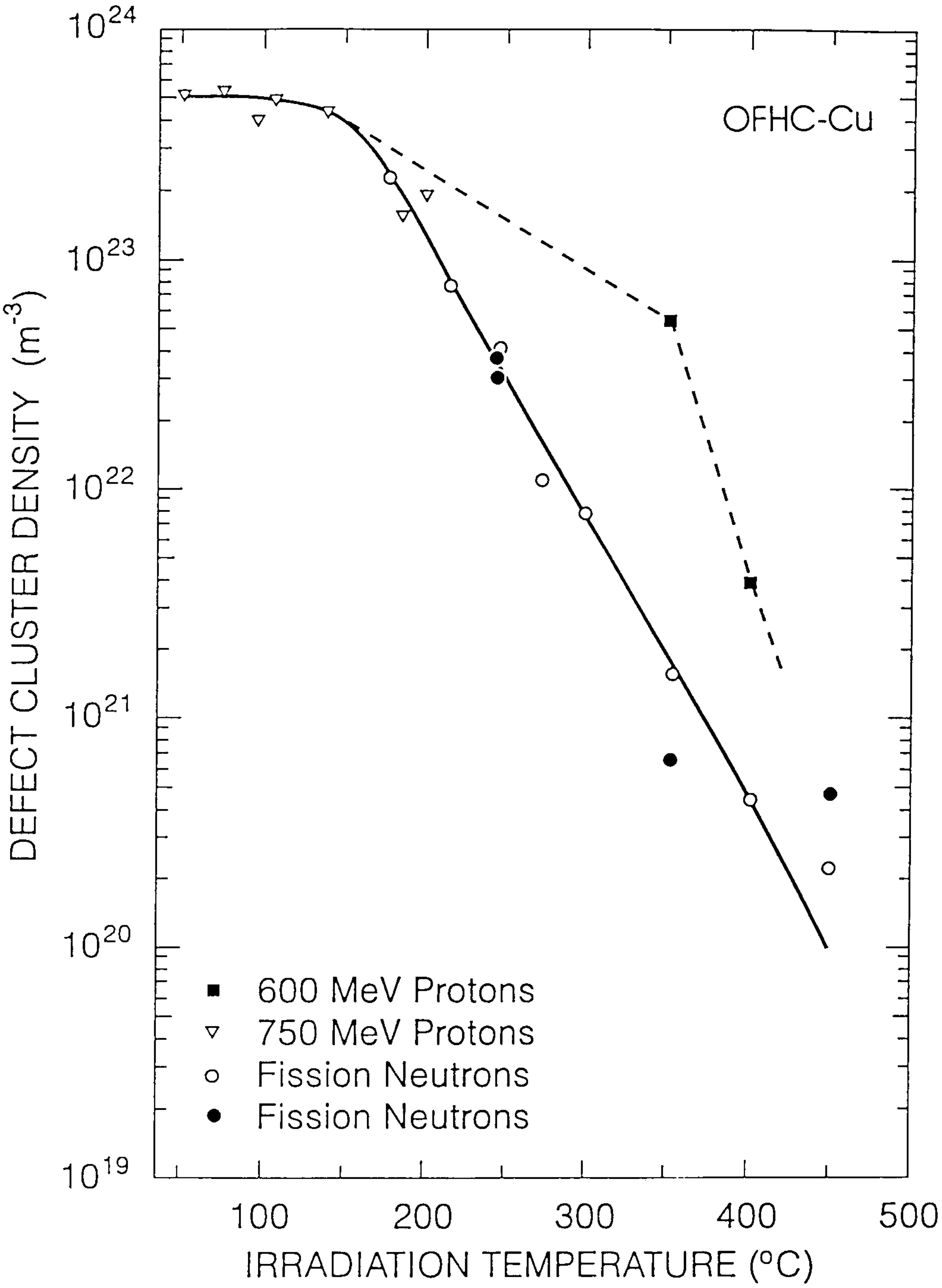

Figure 2. Defect cluster density as a function of irradiation temperature for OFHC-copper irradiated with fission neutrons and 600 and 750 MeV protons to doses between ~0.3 and 1 dpa (NRT). Note that the 600 MeV irradiation yields considerably higher densities of defect clusters than that obtained in specimens irradiated with fission neutrons.

Table 1 (Annex IV): Tensile properties of CuAl-25 irra-diated with 600 MeV protons and fission neutrons.

Irradiation	Sample	Irradiation Temp. [K]	Dose [dpa]	Test. Temp. [K]	$\sigma_{0.2}$ [MPa]	σ_{max} [MPa]	ε_p^u [%]	ε_t [%]	$\varepsilon_{min.}^u$ * [%]
600 Mev Protons	CuAl-25 RCF-2F*	663	~0.5	295	-	450	-	-	5
	CuAl-25 RCF-3F	663	~0.5	295	-	450	-	-	5
	CuAl-25 RCF-4F	663	~0.5	295	-	450	-	-	6
	CuAl-25 RCF-5F	643	~0.5	295	-	450	-	-	6
	CuAl-25 RCF-6F	643	~0.5	295	-	450	-	-	6
	CuAl25 RCF-7F	643	~0.5	295	-	450	-	-	5
Fission Neutrons	CuAl-25 A6-3F	623	~0.3	295	340	460	6.7	20	-
	CuAl-25 A6-5F	623	~0.3	295	340	465	6.8	20	-
	CuAl-25 A1F	723	~0.3	295	340	450	5.7	20	-
	Cu-Al25 A7F	723	~0.3	295	340	440	5.7	20	-

* During tensile testing of RCF specimens there occurred specimen movement in the grips and gave a somewhat inaccurate shape of the stress-strain curves. Hence, neither $\sigma_{0.2}$ nor ε_p^u and ε_t could be obtained reliably. However, σ_{max} values are accurate.

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The present note is the final report on investigations carried out under ITER Task No. T13 EU (1994). Most of the results of these investigations have been published in the open literature either as articles or reports. A list of the appropriate references are given. Results that have been presented at various meetings but have not been published are summarized in the present document. In addition, the present report also clarifies the status of the deliverables in the ITER Task No. T213 EU (1995). Finally, the main conclusions emerging from these investigations are highlighted.

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